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DIELECTRIC RELAXATION IN RARE-EARTH DOPED CALCIUM FLUORIDE CRYS--ETC(U)
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A TRIDENT SCHOLAR
PROJECT REPORT

NO. 88

"DIELECTRIC RELAXATION IN RARE-EARTH
DOPED CALCIUM FLUORIDE CRYSTALS"

UNITED STATES NAVAL ACADEMY
ANNAPOLIS, MARYLAND
1977

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"Dielectric Relaxation in

Rare-Earth Doped

Calcium Fluoride Crystals"

A Trident Scholar Project Report

by

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U. S. Naval Academy

Annapolis, Maryland

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Physics Department

Accepted for Trident Scholar Committee

Chairman

23 May 1977

Date

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ABSTRACT

The complex dielectric constant was measured at five audio frequencies (10^2-10^4 Hz) over a temperature range of 5.5-400K for calcium fluoride crystals doped with 13 different rare-earths of the lanthanide series. The results allow an unambiguous labeling of the spectrum for the first time. Five relaxations were observed which are characteristic of the series and five major new trends were observed in these relaxations. Evidence was also uncovered indicating the possibility of a sixth relaxation occurring at high temperatures.

Work was also done on computer models of a dipolar complex in this crystal which led to the proposal of a new model to account for one of the relaxations. Models were also proposed for the other relaxations observed.

By characterizing the dielectric spectrum for all rare-earth dopants this project has laid the groundwork for a future study on the effects of neutron irradiation of the crystals. It is hoped that this will lead to the development of an improved neutron dosimeter.

ACKNOWLEDGEMENTS

The author would like to thank his advisor, Dr. John Fontanella, for his help in all areas of this project. His continuing amiability, knowledge and support contributed greatly to my enjoyment of and success in the project.

The author would also like to thank Drs. Carl Andeen and Donald Schuele of Case Western Reserve University for the loan of the equipment used in this project.

Thanks are also extended to Fred Wasem for his excellent technical assistance during the course of the project.

Donald J. Link
18 May 1977
Annapolis, Maryland

TABLE OF CONTENTS

I. Introduction

In a pure calcium fluoride crystal the fluorine ions form a simple cubic lattice while the calcium ions form a face-centered cubic lattice. This implies that one-half of the cubes formed by the fluorine lattice are occupied by calcium ions and the other half are unoccupied. When a rare-earth impurity is introduced it is often present as a triply-charged ion, requiring some form of charge compensation in order to preserve charge neutrality. This charge compensation will often result in the formation of dipolar complexes. These dipolar complexes induce a number of electrical relaxations which lend themselves well to analysis because of their Debye-like behavior.

These crystals have received a great deal of attention in recent years. The challenge is to develop a coherent picture of the observed relaxations and develop models of dipolar relaxations to account for their behavior. At the present time only one relaxation has been studied for several rare-earth dopants in calcium fluoride and there are isolated reports of at least five other relaxations in the literature.[1-5] In this project a study was made of the dielectric spectrum of calcium fluoride crystals with thirteen different rare-earth dopants. This allows for an unambiguous labeling of the dielectric spectrum for the

5

first time. Five relaxations are observed that are charac-
teristic of the series with a possible sixth relaxation
occurring at high temperatures. New observations about
their trends of behavior lead to the proposal of models
of dipolar relaxation to account for their behavior.

II. Experiment and Data Reduction

A. Experimental Procedure

The crystals used in the experiment were calcium fluo-
ride crystals with 0.1 mol-% rare-earth impurities. The
rare-earth impurities include the entire lanthanide series
with the exception of prometheum and lanthanum. The
crystals were obtained from Optovac, Inc. in the form of discs
25.4 mm in diameter and about 2.0 mm in thickness. The
crystals were first ground to a thickness of 1.5 mm and
then polished with 1 micron Metadi diamond polishing com-
pound. Aluminum electrodes were evaporated on in the
three-terminal configuration using a shadow ring that gave
a guard gap of .01 mm or less. Good boundary conditions
were ensured by having the ratio of the width of the guard
ring to the thickness of the sample be greater than three.
The crystals were then placed in the eight-sample holder
of a Cryogenics CT-14 cryostat. The temperature range was
from 5.5-400K with an automatic temperature controller
using a platinum resistance thermometer at temperatures
down to 30K and a germanium thermometer at temperatures
below that.

The conductance divided by frequency, G/ω, and the
capacitance, C, were measured using a General Radio 1615
capacitance bridge modified to give meaningful results
for G/ω, and C at levels of less than 1 ppm at the five

audio frequencies 10^2, $10^{2.5}$, 10^3, $10^{3.5}$, and 10^4 Hz.
The generator-detector assembly was designed to allow
single-knob switching between frequencies. An interface
was designed to give a digital readout after the bridge
was manually balanced.

B. Analysis of Data

The real part of the dielectric constant, ϵ', was set
equal to 6.915 at 300K and 1000Hz for all 0.1 and 1.0 mol-%
samples. This is the value given by the two-fluid tech-
nique for calcium fluoride with a 0.1 mol-% erbium impu-
rity.[3] This was done for two reasons; first, the differ-
ence between this value and the correct value is negli-
gibly small, and second, most of the important results are
derived from relative changes in ϵ' and ϵ'' so a change in
the absolute value of the entire curve would have minimal
effect on the results.

To determine ϵ' for other frequencies at 300K the
relationship: $\epsilon'/\epsilon'_{1000Hz} = C/C_{1000Hz}$ was used. ϵ' at
temperatures other than 300K was determined from:

$$\frac{\epsilon'_T}{\epsilon'_{300}} = \frac{C_T}{C_{300}} \; EXP(-\int_{300}^{T} \alpha_p dT) \tag{1}$$

where α_p is the isobaric linear thermal expansion coef-
ficient. Since α_p is unknown for rare-earth doped samples
the value of α_p for pure calcium fluoride was used.[6]

Then the imaginary part of the dielectric constant at
each temperature and frequency was calculated using the
equation:

$$\epsilon_T'' = \frac{\epsilon_T'' G}{\omega C} \tag{2}$$

Figure 1 contains the graphs of ϵ'' vs. T at 100 Hz for 0.1 mol-% impurities of thirteen different rare-earths. They are listed in order of increasing atomic number (and decreasing ionic radius) starting with Ce and going down to Gd on the left and continuing with Tb through Lu on the right. For the other four frequencies the plots are basically the same except that the peaks occur at succesively higher temperatures as the frequency increases. It is readily apparent from figure 1 that there are certain peaks which are characteristics of the series. These peaks will be discussed individually in a later section.

Next, at each of the five frequencies the peaks were fit to the Debye equation:

$$\epsilon'' = \frac{A\omega\tau}{T(1+\omega^2\tau^2)} \tag{3}$$

in order to obtain the temperature at which $\omega\tau = 1$. T is the absolute temperature, τ is the relaxation time, and A is the dipole strength, given by:

$$A = \frac{Np^2}{3\epsilon_o k} \tag{4}$$

where N is the dipole concentration, p is the dipole moment, ϵ_o is the permittivity of free space and k is Boltzmann's constant. A is assumed to be constant for a given peak.

The relaxation time, τ, is given by an Arrhenius equation of the form:

$$\tau = \tau_0 EXP(E/kT) \tag{5}$$

where τ_0 is the reciprocal frequency factor and E is the activation energy. Equation (5) and $\omega\tau = 1$ imply:

$$\ln(\omega) = -E/kT - \ln(\tau_0) \tag{6}$$

Finally, the five sets of (ω, T) obtained from equation (3) were fit into equation (6) to give the values of E and τ_0 for individual peaks. Table 1 gives a listing of activation parameters for the peaks for which fits were obtainable. The activation energy is an almost linear function of the peak position. If an accurate fit of the peak can't be made because of background, a reasonable estimate of the activation energy can be made based on the peak position.

C. Computer Simulation

Work was also done on designing computer models of the dipolar complex shown in figure 3. The first program was designed to give the difference in potential energy between various sites for an interstitial fluorine ion. The model included the effects of short range repulsion (using the short range repulsion parameters for the F^--F^- and $Ca^{2+}-F^-$ interactions given by Wong[7]) and electrostatic interaction using the Ewald/Evjen method and including ions out to the sixth nearest neighbor. Due to lack of availability of short range repulsion parameters for

RE^{3+}-F^- interactions, the Ca^{2+}-F^- parameters were used.

Next, work was done on a computer model to discover how the lattice would relax as the interstitial fluorine ion moved through the saddle point between two equilibrium positions. The technique used was similar to that used for the first program, but the short range repulsion parameters for the RE^{3+}-F^- interactions were varied to simulate the varying sizes of the rare-earth ions. The program is listed in Appendix.A.

III. Discussion of Results

A. R_I Relaxation

The central peak, at about 200K with an activation energy of about .4 eV as seen in table 1, has been the most widely studied. It is the only peak for which a relatively reliable ESR correlation has been made. ESR line broadening experiments show a 0.4 eV process with tetragonal symmetry. The simplest model to account for the known behavior of this peak would be charge compensation by a nearest-neighbor interstitial fluorine ion (as is seen in figure 3). Figure 2 is a graph of ε'' vs. concentration and temperature for 0.1 mol-% Er from a paper by Andeen and Fontanella[3]. As can be seen from the graph, the height of the R_I peak at first increases with increasing concentration, reaches a peak at a concentration of about 0.1 mol-%, and then decreases.

An explanation for this is that as the concentration in-
creases the number of complexes of this type at first in-
creases, but as the concentration gets too high the crystal
becomes crowded with rare-earth ions and clusters pre-
dominate.

The activation parameters for this peak compare rea-
sonably well with the values recently obtained by Kitts
and Crawford using the ITC technique[1]. However, the first
main result of this study indicates that the activation
energy is relatively independent of rare-earth ionic radius
rather than obtaining a slight increase in activation
energy as ionic radius increases as reported in their
paper. It is quite possible that their results for Tm
and Yb are too low because of the non-Debye-like behavior
of the R_I peaks for Tm, Yb, and Lu. Analysis of present
data indicated that this peak is indeed much broader than
for the other rare-earths so no activation parameters were
obtained for this peak for Tm, Yb, and Lu in the present
work.

As mentioned above, this peak has been traditionally
associated with the relaxation of a nearest-neighbor
fluorine ion as shown in figure 3. This model is suppor-
ted by the ESR correlation and concentration dependence
of the relaxation. However, it is disturbing that a
relaxation that occurs so close to a rare-earth ion
should have an activation energy so independent of ionic
radius.

B. R_{II} Relaxation

The second major result of this work is the discovery
that the R_{II} peak disappears for all rare-earths with an
ionic radius larger than that of Gd^{3+}. It can also be seen
that the size of the peak increases monotonically with
decreasing ionic radius. As can be seen from figure 2
this peak has approximately the same dependence on concen-
tration as the R_I peak, possibly indicating that the asso-
ciated complexes for both of the peaks have the same num-
ber of rare-earth ions. It was difficult to obtain acti-
vation parameters because of the complication introduced
by the presence of other peaks, but it can be seen from
the positions of the peaks in figure 1 that, with the ex-
ception of Lu, the activation energy decreases with decrea-
sing rare-earth ion size. Several mechanisms have been
proposed for this relaxation, none of which appears to be
particularly reliable.

One possible new model for this relaxation is the
relaxation of the nearest-neighbor fluorine ion by a dif-
ferent mode than that for the R_I peak. There have been
two possible modes of relaxation proposed for the relaxa-
tion of a nearest-neighbor fluorine. Figure 3 shows the
direct jump mode, in which the interstitial ion moves
directly from one position to another by a route between
the two lattice fluorines and a neighboring calcium ion.
Figure 4 illustrates the interstitialcy mode, in which

a lattice fluorine moves into the interstitial position
and is replaced by the old interstitial ion. The results
of the first computer model (unrelaxed lattice) discussed
in section II.C. indicated the possibility of a third mode
of relaxation, such as is shown in figure 5. In this
mode of relaxation the fluorine ion would move directly
from one position to another as in the direct jump mode,
but via a path between the two lattice fluorines and the
rare-earth ion rather than the calcium ion. The results
of the the second computer model (relaxed lattice) indi-
cate that this mode of relaxation is not possible if the
short range repulsion parameters for the RE^{3+}-F^- inter-
action are too high. Because of lack of data on the
short range repulsion parameters for the RE^{3+}-F^- inter-
action it was not possible to determine for which of the
rare-earth ions this mode of relaxation would disappear.
It is possible that the R_{II} peak is due to this mode of
relaxation and that the R_I peak is due to one of the other
modes of relaxation. This would account for the larger
dependence of the activation energy on rare-earth ionic
radius for the R_{II} peak in comparison with the R_I peak.
This would also account for the fact that both of the
peaks have approximately the same dependence on concen-
tration.

There are three major shortcomings to this proposal.
In the unrelaxed lattice computer model it was found that
the activation energy for the mode of relaxation shown

in figure 5 is less than the activation energy for the mode of relaxation shown in figure 3. however it must be expected that in a relaxed lattice the activation energy would be larger for the former mode of relaxation. or else it would not disappear with too large a short range repulsion parameter for the RE^{3+} - F^- interaction. So it would be expected that this peak would have a larger activation energy than the R_I peak, which is not the case. Also, if both of these peaks were due to a single dipolar complex, the peak occurring at a higher temperature would not be expected to appear in ITC data. Once again this is not the case. Finally, it appears that the peaks do not anneal together; the R_{II} peak increasing approximately three times as much as the R_I peak after annealing.

C. R_{III} Relaxation

The third major result of this study is that the R_{III} peak, discovered recently by Fontanella, et al[3] disappears for rare-earths with a larger ionic radius than that of Tb^{3+}. Because of the irregularity of the peak few activation parameters could be obtained. From figure 2 it can be seen that this peak has a very strong dependence on concentration, stronger than for any other peak. Based on this it is generally accepted that this peak is due to a cluster complex, possibly involving four or more rare-earth ions.

D. R_{IV} Relaxation

The fourth major result of this work is the discovery of a relaxation with an activation energy which depends strongly on the nature of the rare-earth. In fact, the dependence is almost linear, as is shown in figure 6, a graph of activation energy versus rare-earth ionic radius. Figure 2 shows that this peak has a higher dependence on concentration than the R_I or R_{II} peaks, but not as high a dependence on concentration as the R_{III} peak. Consequently the model proposed for this relaxation involves two rare-earth ions, as is shown in figure 7. The extra fluorine ion required for charge neutrality would have to be trapped elsewhere in the lattice. The fact that the fluorine ion has to pass between two rare-earth ions in order to relax would account for the strong dependence of the activation energy on the rare-earth ionic radius. A second way to account for this strong dependence would be a complex in which the rare-earth ion itself relaxed.

E. R_V Relaxation

The fifth major result of this project is the discovery of an additional relaxation which is a characteristic of the series. Little can be said about this peak other than what can be seen in figure 1. This peak is similar to the R_{IV} peak in its strong dependence of

16

activation energy on rare-earth ion size although the dependence is not as large.

F. High Temperature Tail

Another result of this project is the discovery that the high temperature tail commences at increasingly lower temperatures as the ionic radius of the rare-earth dopant decreases. In the past this feature has commonly been associated with DC conductivity, a phenomenon that is only indirectly associated with defect centers. However the excessively strong dependence of this peak on the rare-earth dopant indicates the possibility that this tail may be due to the beginning of another peak, possibly associated with the relaxation reported by Franklin, et al, with an activation energy of 1.2 eV.[5] The position of the peak would indicate an activation energy in close agreement with this figure.

G. Practical Applications

The original purpose of this project was to find a crystal with a zero temperature coefficient for use as the transducer in a high pressure gauge. However, it was soon discovered that these crystals were not suitable for that purpose and attention was directed toward another use of these crystals. It was hoped that the main achievement of this project, the characterization of the dielectric spectrum for almost all rare-earths of the lanthanide series,

would lay the groundwork for a study on the effects of neutron irradiation of these crystals.

The rare-earths have a large absorption cross section, particularly Gd and Eu. So it is expected that neutron irradiation of these crystals will have a large effect on them. (It has already been shown that the R_{II} peak can be grown in a Eu doped sample by gamma irradiation[3].) And based on the characterization of the peaks provided by this project, it may be possible to determine which new rare-earths are introduced as decay products. The R_{IV} peak, which occurs at different temperatures for different dopants, will be particularly useful in this work. Midshipman Kolodziejzak has proposed a Trident Project to study the effects of neutron irradiation of these crystals next year. It is hoped that this will lead to the development of an improved neutron dosimeter.

IV. Conclusion

The five major results of this work are the discovery of the independence of the activation energy with respect to rare-earth for the R_I peak, the disappearance of the R_{II} and R_{III} peaks for the lower numbered rare-earths, the extremely strong dependence of activation energy on rare-earth ion size for the R_{IV} peak, and the discovery of a new relaxation which is a characteristic of the series. Another result is the discovery of the movement of the high temperature tail as a function of the rare-earth dopant, leading to the possibility that it may be due to another relaxation.

However the main achievement of this project is the characterization of the dielectric spectrum for all rare-earth dopants. This characterization has laid the groundwork for a study on the effects of neutron irradiation of the crystals. It is hoped that this will lead to the development of an improved neutron dosimeter.

Table I. Activation parameters for various relaxations in rare-earth doped calcium fluoride.

	R_I Relaxation		R_{II} Relaxation		R_{III} Relaxation		R_{IV} Relaxation	
	E(ev)	τ_0(10^{-14} sec)	E(ev)	τ_0(10^{-13} sec)	E(ev)	τ_0(10^{-12} sec)	E(ev)	τ_0(10^{-15} sec)
Ce	.431	1.39						
Pr	.432	1.29					.91	
Nd	.429	1.42					.89	
Sm	.403	2.41					.793	2.47
Eu	.414	1.59					.757	2.37
Gd	.414	1.68					.723	2.59
Tb	.404	1.88			.046	1.22	.645	5.35
Dy	.406	1.85					.604	7.64
Ho	.409	1.74	.151	2.04	.029	8.67	.580	6.19
Er	.406	1.88	.146	1.91	.028	10.7	.543	5.51
Tm			.133	2.64			.48	
Yb			.119	4.72				
Lu			.128	2.08	.031	2.54		

Figure 1. ϵ'' vs T for 13 rare-earth impurities
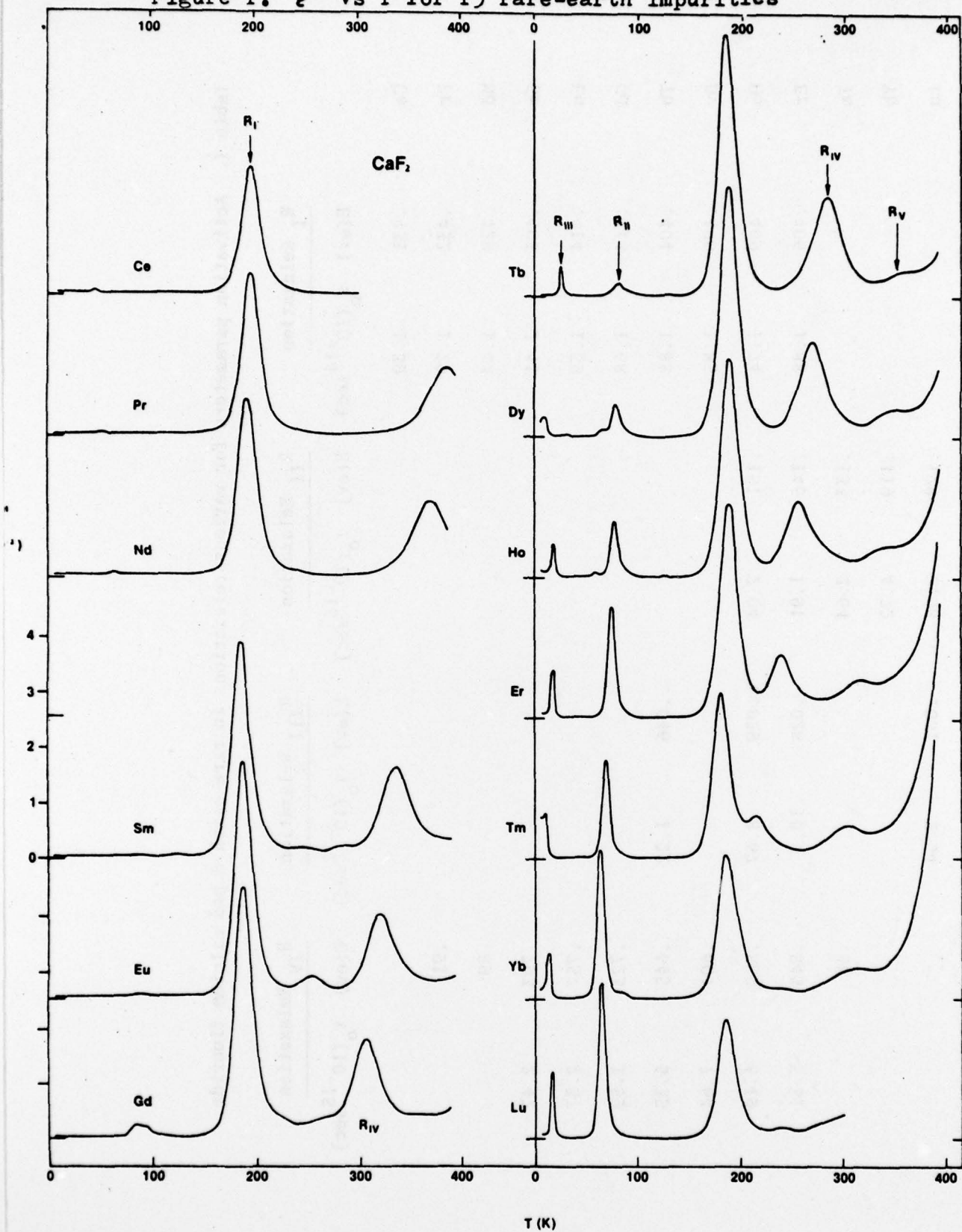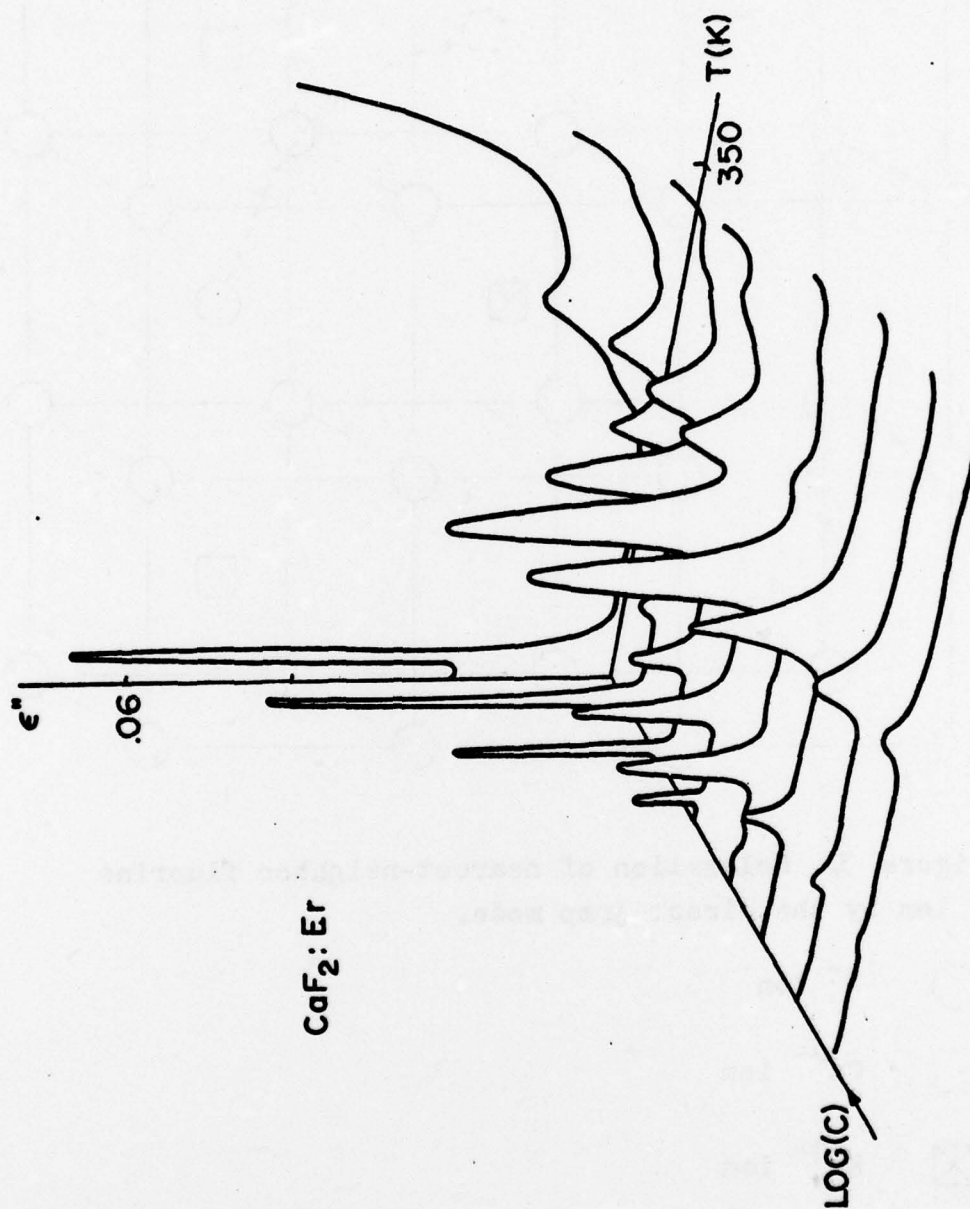
Figure 2. Imaginary part of the dielectric constant, ε'', at 1000 Hz vs. temperature for all eight concentrations of CaF_2:Er^{3+}.
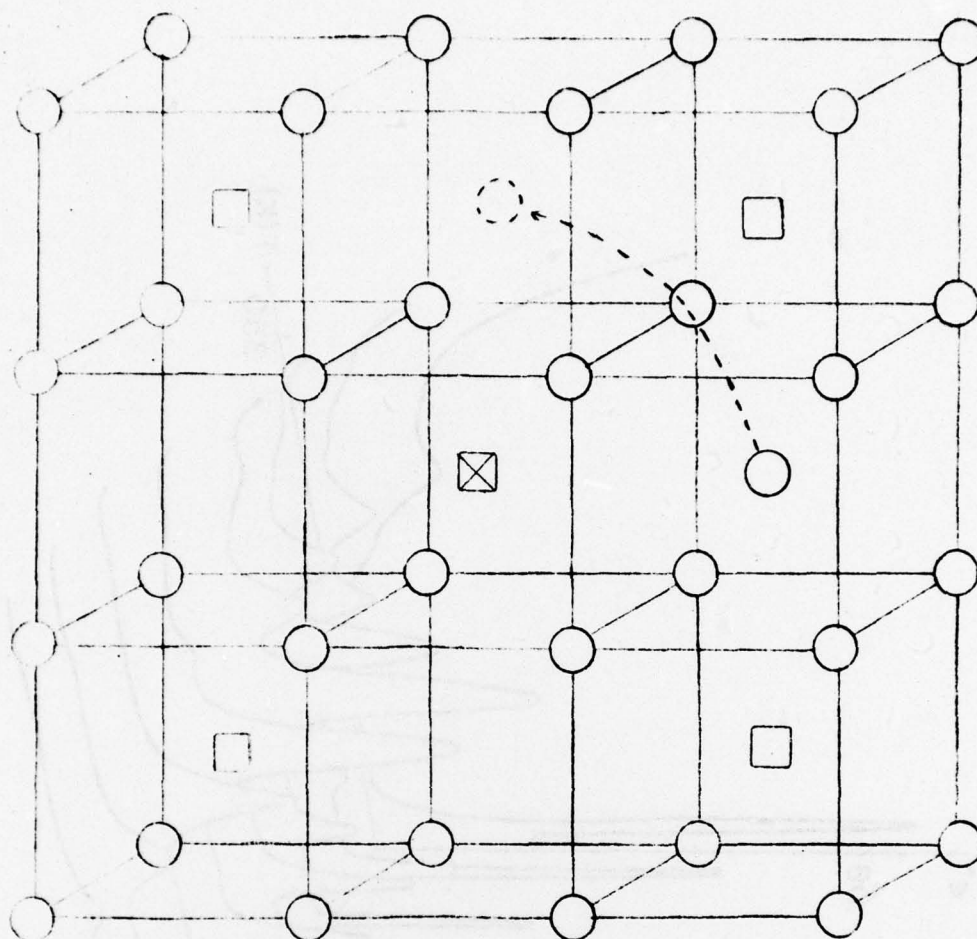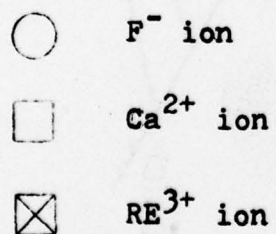
Figure 3. Relaxation of nearest-neighbor fluorine
   ion by the direct jump mode.

$\bigcirc$ F^- ion

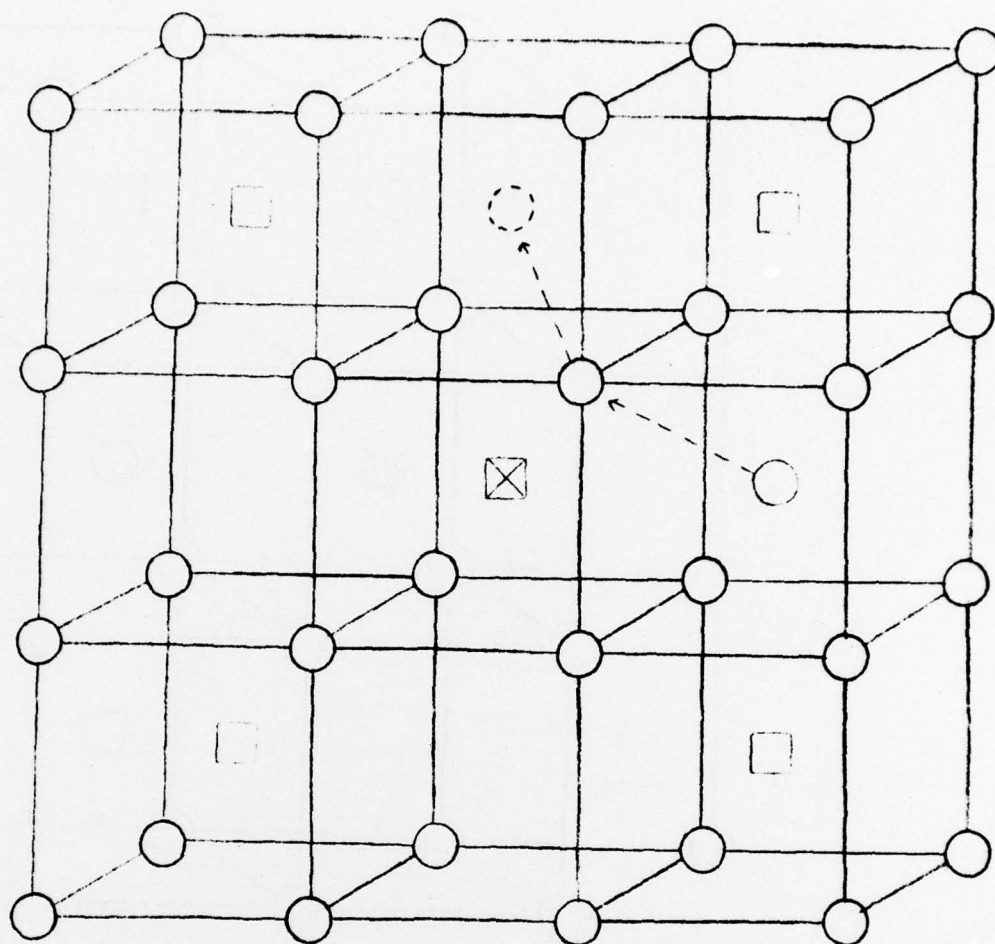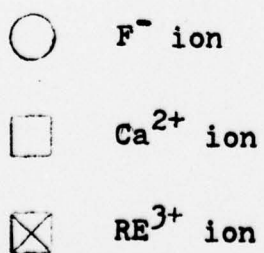$\square$ Ca^{2+} ion

$\boxtimes$ RE^{3+} ion

Figure 4. Relaxation of the nearest-neighbor
            fluorine ion by the interstitial mode.

$\bigcirc$ F^- ion

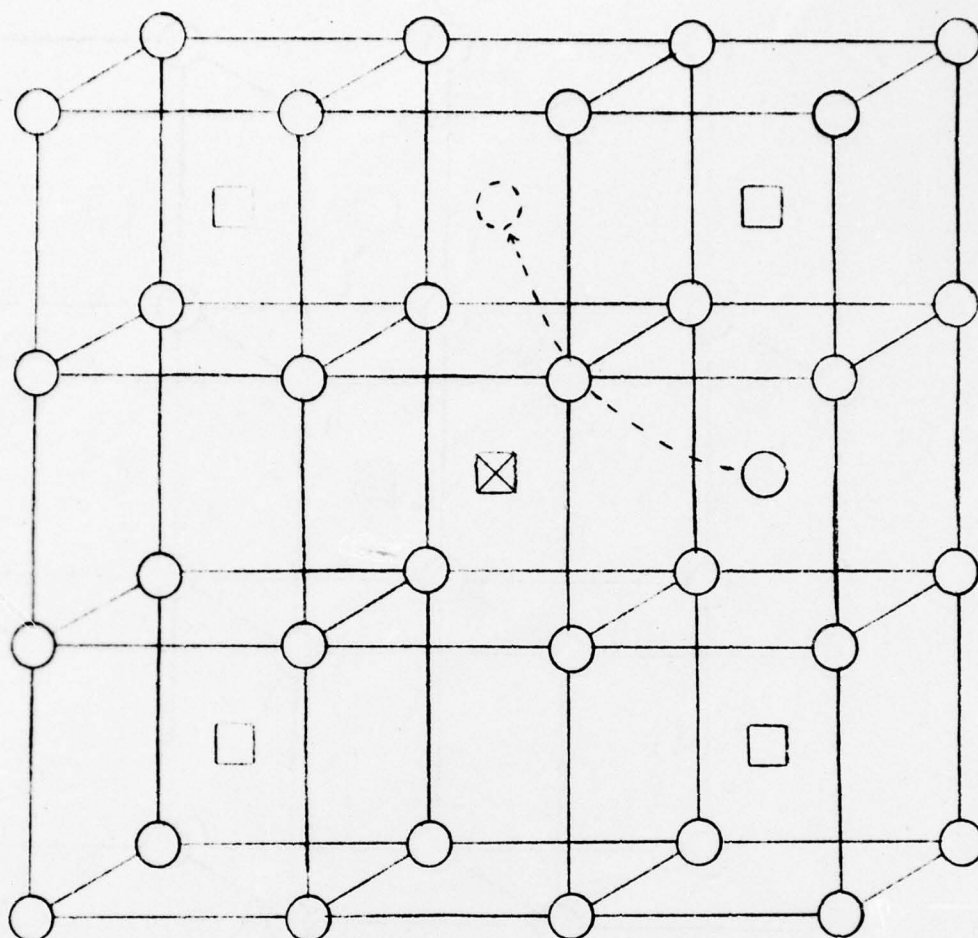$\square$ Ca^{2+} ion

$\boxtimes$ RE^{3+} ion

Figure 5. Relaxation of the nearest-neighbor
fluorine ion by the alternate direct
jump mode.

○ F^- ion

□ Ca^{2+} ion

⊠ RE^{3+} ion

E (ev)

1.

0.88

0.76

0.64

0.52

0.4

Tm

Er

Ho

Dy

Tb

Gd

Eu

Sm

Nd

Pr

R_{IV} RELAXATION

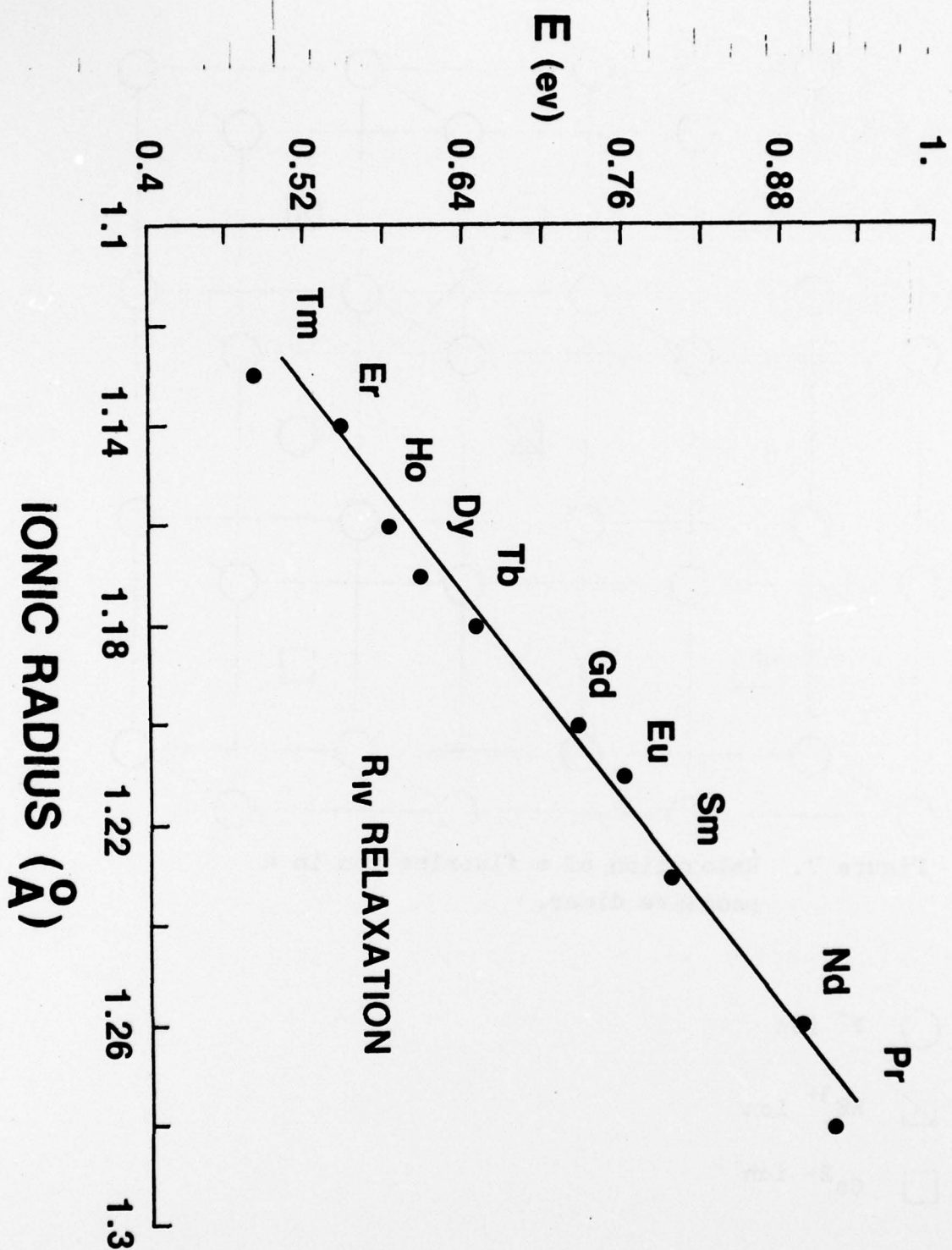1.1 1.14 1.18 1.22 1.26 1.3

IONIC RADIUS (Å)

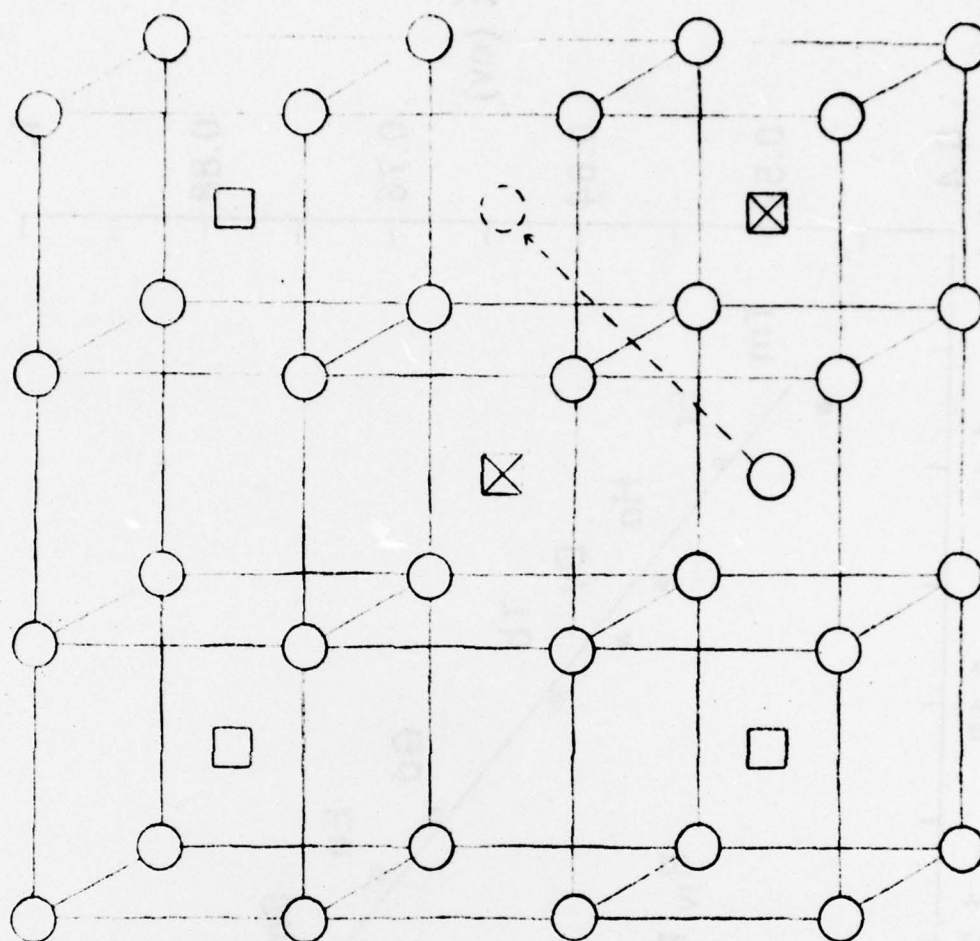Figure 6. Activation energy vs ionic radius

Figure 7.   Relaxation of a fluorine ion in a
            positive dimer.

○   F⁻ ion

⊠   RE³⁺ ion

□   Ca²⁺ ion

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APPENDIX A

```
100)DIMENSION A(-6:6,-6:6,-6:6),B(-6:6,-6:6,-6:6),C(-6:6,-6:6,-6:6)
110LOGICAL Q
120Z1=.85372E+5
130Z2=-9.4014
140Y1=.96965+3
150Y2=-13.293
160G=31.031
161G2=-4.5
162S1=1E+6
163 15 U=1.4
164X=0.
165 13 D=.2
170DO 1 I=-6,6
180DO 1 J=-6,6
190DO 1 K=-6,6
200A(I,J,K)=I
210B(I,J,K)=J
220 1 C(I,J,K)=K
230A(2,0,0)=B(2,0,0)=U
270R=10.
290 11 D=.85*D
300 Q=.FALSE.
310DO 2 L=-1,3
320DO 2 M=-1,3
330DO 2 N=-2,2
350T1=T2=T3=0.
360IF (100*L+10*M+N .NE. 0) GO TO 7
370DO 3 I=-6,6
380DO 3 J=-6,6
390DO 3 K=-6,6
400IF (100*I+10*J+K .EQ. 0) GO TO 3
410Z3=(A(L,M,N)-A(I,J,K))^2+(B(L,M,N)-B(I,J,K))^2+(C(L,M,N)-C(I,J,K))^2
420Z=SQRT(Z3)
430F=0.
440IF(MOD(I*J*K,2).NE.0 .OR. 100*I+10*J+K.EQ.200) F=-3*G/Z3+S1*EXP(S2*Z)
450IF (MOD(I+J+K,4).EQ.0 .AND. MOD((I+1)*(J+1)*(K+1),2).NE.0) F=6*G/Z3
460IF(I .EQ. -6 .OR. I .EQ. 6)F=F/2
461IF(J .EQ. -6 .OR. J .EQ. 6)F=F/2
462IF(K .EQ. -6 .OR. K .EQ. 6)F=F/2
470T1=T1+F*(A(L,M,N)-A(I,J,K))/Z
480T2=T2+F*(B(L,M,N)-B(I,J,K))/Z
490T3=T3+F*(C(L,M,N)-C(I,J,K))/Z
500 3 CONTINUE
510GO TO 4
520 7 IF(MOD(L*M*N,2) .NE. 0 .OR. 100*L+10*M+N .EQ. 200) GO TO 5
530IF (MOD(L+M+N,4).NE.0 .OR.MOD((L+1)*(M+1)*(N+1),2).EQ.0) GO TO 2
540DO 6 I=-6,6
```

```
100 DIMENSION A(-6:6,-6:6,-6:6),B(-6:6,-6:6,-6:6),C(-6:6,-6:6,-6:6)
110 LOGICAL Q
120 Z1=.85372E+5
130 Z2=-9.4014
140 Y1=.9696E+3
150 Y2=-13.293
160 G=31.031
161 G2=-4.5
162 S1=1E+6
163 15 U=1.4
164 X=0.
165 13 D=.2
170 DO 1 I=-6,6
180 DO 1 J=-6,6
190 DO 1 K=-6,6
200 A(I,J,K)=I
210 B(I,J,K)=J
220 1 C(I,J,K)=K
230 A(2,0,0)=B(2,0,0)=U
270 R=10.
290 11 D=.85*D
300 Q=.FALSE.
310 DO 2 L=-1,3
320 DO 2 M=-1,3
330 DO 2 N=-2,2
350 T1=T2=T3=0.
360 IF (100*L+10*M+N .NE. 0) GO TO 7
370 DO 3 I=-6,6
380 DO 3 J=-6,6
390 DO 3 K=-6,6
400 IF (100*I+10*J+K .EQ. 0) GO TO 3
410 Z3=(A(L,M,N)-A(I,J,K))^2+(B(L,M,N)-B(I,J,K))^2+(C(L,M,N)-C(I,J,K))^2
420 Z=SQRT(Z3)
430 F=0.
440 IF(MOD(I+J+K,2).NE.0 .OR. 100*I+10*J+K.EQ.200) F=-3*G/Z3+S1*EXP(S2*Z)
450 IF (MOD(I+J+K,4).EQ.0 .AND. MOD((I+1)*(J+1)*(K+1),2).NE.0) F=6*G/Z3
460 IF(I .EQ. -6 .OR. I .EQ. 6)F=F/2
461 IF(J .EQ. -6 .OR. J .EQ. 6)F=F/2
462 IF(K .EQ. -6 .OR. K .EQ. 6)F=F/2
470 T1=T1+F*(A(L,M,N)-A(I,J,K))/Z
480 T2=T2+F*(B(L,M,N)-B(I,J,K))/Z
490 T3=T3+F*(C(L,M,N)-C(I,J,K))/Z
500 3 CONTINUE
510 GO TO 4
520 7 IF(MOD(L+M+N,2) .NE. 0 .OR. 100*L+10*M+N .EQ. 200) GO TO 5
530 IF (MOD(L+M+N,4).NE.0 .OR.MOD((L+1)*(M+1)*(N+1),2).EQ.0) GO TO 2
540 DO 5 I=-6,6
```

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The study was made of the dielectric spectrum of calcium fluoride crystals with thirteen different rare-earth dopants.

The paper describes the technical procedures of the experiment and results. The work was also done on computer.

Five relaxations were observed which are characteristics of the series, with a possible sixth relaxation occuring at high temperatures. However the main achievement of this project is the characterization of the dielectric

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spectrum for all rare-earth dopants. This characterization has laid the groundwork for a study on the effects of neutron irradiation of the crystals. The writer hopes that this will lead to the development of an improved dosimeter. -

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